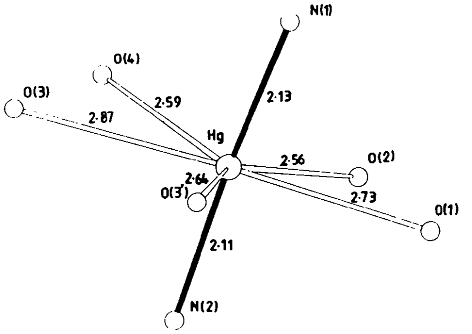

Fig. 2. Coordination around Hg. All e.s.d.'s 0·02 Å. O(3′) is at $1-x, 1-y, 1-z$.

range (Grdenić, 1965), the direction of the bonds is slightly but significantly non-linear. Similar small departures from linearity are observed in most Hg compounds which are primarily two-coordinated. Two O atoms from each trifluoroacetate residue, O(1), O(2), O(3), O(4), and one, O(3′), from a centrosymmetrically related trifluoroacetate, complete the coordination (Fig. 2). The distances of these atoms from the best least-squares plane through them and the Hg are respectively 0·021, −0·083, −0·142, 0·163, 0·059 and (for Hg) −0·019 Å. The angles between this plane and Hg—N(1), Hg—N(2) are respectively 80·1 and 90·0°. O(3) is bound to two Hg atoms. The Hg—O distances are considerably longer than the typical covalent distance (Grdenić, 1965) and must be regarded as being secondary in character. The disposition of the trifluoroacetate groups and the lengths of the C—O bonds (all within the range 1·20 to 1·25 Å) suggest that these groups are ionic with the group $Hg(py)_2^{2+}$ acting as the cation. The presence of trifluoroacetate ions was confirmed by the IR spectrum of the solid material.

The pyridine residues are planar to within the limits of error and the distances and angles are those normally found. The two pyridine rings are twisted about the N—N vector and make an angle of 31·1° to each other. The $Hg(py)_2^{2+}$ ion is similar to that reported in bis(pyridine)mercury(II) nitrate dihydrate (Kamenar, Hergold-Brundić & Sikirica, 1976), where the Hg—N distances are 2·129 (8) and 2·135 (8) Å, the angle N—Hg—N is 176·8 (3)° and the overall coordination is described as distorted octahedral with only four O atoms in the equatorial plane. Seven-coordinated Hg, similar to that found in the title compound, has been observed in tetraethyleneglycol(dimethyl ether) mercury(II) chloride (Iwamoto, 1973) in which there are two axial Cl atoms at 2·31 and 2·29 Å and five equatorial O atoms at 2·96, 2·78, 2·79, 2·82 and 2·86 Å. In the trifluoroacetate ions of the present compound, the atoms O, O, C, C are coplanar within the limits of error and there is evidence of considerable thermal vibration of the CF_3 groups of both ions. Apart from the Hg—O bond referred to above, there is no evidence of intermolecular bonding between adjacent units of the structure.

We thank the Science Research Council for the provision of a research studentship to one of us (JH).

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2-*exo*-(Chloromercurio)-3-*exo*-acetoxybicyclo[2.2.1]heptane

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Abstract. $C_9H_{13}ClHgO_2$, monoclinic, $P2_1/c$, a = 14·10 (1), b = 10·48 (1), c = 7·218 (7) Å, β = 96·33 (7)°, with $Z = 4$, $D_c = 2{\cdot}44$, $D_m = 2{\cdot}38$ g cm^{-3}. Crystals were invariably twinned about c. The structure was determined from 423 diffractometer-measured intensities (Mo $K\alpha$, λ = 0·7107 Å) which were free from twinning overlap, R = 0·080. The distances Hg—Cl and Hg—C are 2·32 (2) and 2·10 (6) Å, the

angle Cl—Hg—C is 173 (2)°. The results indicate that the compound has been formed by *cis* addition across the norbornene ethylenic bond.

Introduction. The structures of the products of oxymercuration of olefins are of interest in that they provide valuable information concerning the stereochemistry of addition reactions. It is now believed that the reactions normally proceed by *trans* addition, but *cis* addition occurs if the starting material contains a strained double bond as in norbornene (bicyclo[2.2.1]heptene).

Although the structure of the oxymercuration products of cyclohexene have been established by NMR (Anderson & Henry, 1961) and by crystallographic methods (Brook & Wright, 1951), corresponding crystallographic studies have not been reported for the oxymercuration products of norbornene. Structures of some products have been deduced by spectroscopic methods; for example Anderson & Henry (1961) suggested a *cis-exo* configuration for 2-hydroxy-3-(chloromercurio)norbornane on the basis of its NMR spectrum. Traylor & Baker (1963), relying mainly on IR spectroscopy, have proposed a similar structure for 2-hydroxy-3-(acetoxymercurio)norbornane. The work reported here seeks to confirm these conclusions.

The title compound was prepared by the method of Tobler & Foster (1965) and recrystallized from petroleum ether. Attempts to prepare single crystals were unsuccessful; acicular crystals twinned about the needle axis, *c*, were invariably obtained, the twins were inseparable and their presence was shown by Weissenberg photographs and confirmed with the polarizing microscope. Twinned crystals from acetone were used for all measurements; the cell dimensions and space group were determined from Weissenberg photographs using Cu $K\alpha$ radiation ($\lambda = 1{\cdot}5418$ Å).

Using crystals set respectively about *c* and *b*, intensities from one twin (where possible) were measured using the Stoe STADI-2 automatic two-circle diffractometer and graphite-monochromatized Mo $K\alpha$ radiation. As with most Hg compounds, irradiation produced progressive decomposition accompanied by broadening of the reflexions. On account of this broadening and the proximity of twinned reflexions, it was impossible to choose scanning ranges which would either resolve individual reflexions or combine the effects of both twins, for all reflexions. With *c* as the twinning axis, reflexions $hk0$ from the chosen twin are superimposed exactly upon reflexions $\bar{h}\bar{k}0$ from the other twin; using the *c* axis crystal, the combined intensities were measured. The relative weights of the two twins were established by measuring on the first layer line the strongest $hk\bar{1}$ reflexions from the second twin in addition to $hk1$ from the chosen one. On account of partial overlap, no attempt was made to measure intensities for reflexions of type $hk2$ and $hk5$ for either setting axis; in addition $hk0$ reflexions were omitted for the *b* axis crystal. A comparison of reflexion angles for the two twins over the remaining reflexions was made and any reflexions lying within 2° of one from the other twin were deleted from the data. With a maximum 2θ of 40° there remained 347 significant reflexions about *c* and 423 about *b* after elimination of those for which $I < 3\sigma(I)$. The average crystal dimensions were $0{\cdot}01 \times 0{\cdot}1 \times 0{\cdot}4$ mm (μ for Mo $K\alpha = 141{\cdot}9$ cm^{-1}).

Table 1. *Mean fractional atomic coordinates* ($\times 10^3$)

	x	*y*	*z*
Hg	102 (0)	577 (0)	710 (0)
Cl	24 (1)	687 (2)	461 (2)
C(1)	173 (4)	457 (4)	911 (8)
C(2)	284 (4)	464 (5)	919 (9)
C(3)	161 (4)	501 (5)	116 (9)
C(4)	310 (4)	501 (6)	128 (11)
C(5)	232 (3)	599 (4)	162 (8)
C(6)	184 (4)	394 (6)	244 (10)
C(7)	290 (4)	396 (5)	245 (10)
O(1)	313 (3)	559 (3)	805 (6)
C(8)	401 (5)	543 (6)	731 (10)
O(2)	440 (4)	440 (4)	767 (7)
C(9)	427 (4)	644 (6)	617 (10)

Absorption corrections were calculated using the suite of programs *SHELX* (Sheldrick, 1976) which also performed data reduction and produced a single set of F_o values from the two sets of intensity measurements. Hg and Cl atom positions were derived from Patterson maps; on these, and on Fourier maps phased on Hg positions, there were satellite peaks which were ascribed to incomplete series effects arising from the omission of all reflexions for $l = 2$, $l = 5$. This was confirmed by $F_o - F_c$ Fourier maps which did not show these satellites but revealed the positions of the remaining non-H atoms.

Refinement was carried out using the full-matrix least-squares program *CRYLSQ* of the XRAY 72 system (Stewart, Kruger, Ammon, Dickinson & Hall, 1972). Only Hg and Cl atoms were given anisotropic thermal factors. An improvement in the agreement was found to result from the inclusion of H atoms at their expected positions. Using the combined cross-scaled data from both axes the value of R was 0·082. The *c* and *b* axis data refined separately using inter-layer scale factors gave $R = 0{\cdot}069$ and $R = 0{\cdot}080$.

Atomic scattering factors for C, O and H were taken from *International Tables for X-ray Crystallography* (1962) and for Hg atoms from Cromer & Mann (1968). Final atomic positions (mean values) are given in Table 1.*

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* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33696 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

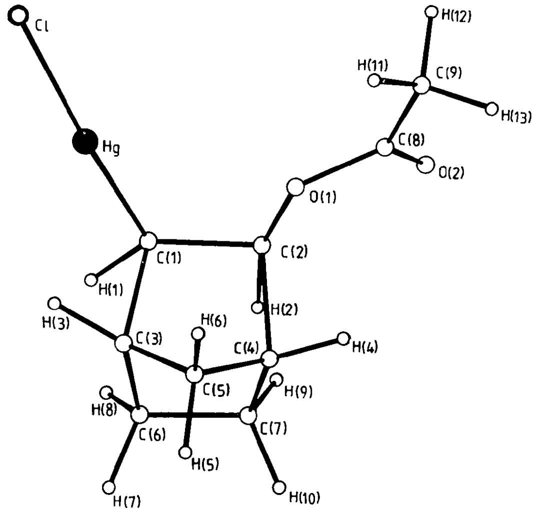

Fig. 1. A view of the $C_9H_{13}ClHgO_2$ molecule, showing the atom numbering scheme used in this analysis.

Discussion. An illustration of the molecule drawn using the program *ORTEP* (Johnson, 1970) is given in Fig. 1.

The Hg atom is bonded almost linearly [173 (2)°] to Cl and C(1); the bond lengths 2·32 (2), 2·10 (6) Å are within the range most frequently found for covalent Hg—Cl and Hg—C distances. Similar small deviations from linearity are typically found in two-coordinate Hg compounds. The nearest other contacts to the Hg atoms are Cl atoms of adjacent molecules (3·43 and 3·32 Å); these are in excess of the sum of the van der Waals radii, r_{Cl} 1·80 Å (Pauling, 1940) and r_{Hg} 1·50 Å (Grdenić, 1965), and cannot be ascribed to definite interactions. The small torsion angle for Hg—C(1)—C(2)—O(1) of 7 (5)° indicates that addition across the norbornene ethylenic bond is in the *cis* conformation, confirming the prediction of Anderson & Henry (1961) and of Traylor & Baker (1963). The bond distances and angles in the norbornane nucleus agree, within the limits of experimental error, with those in similar compounds.

We thank the Science Research Council for the provision of a Research Studentship to one of us (JH).

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Cobalt Dihydrogen Diphthalate Hexahydrate

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Abstract. $Co(C_8H_5O_4)_2.6H_2O$, monoclinic, $P2_1/c$; $a = 6{\cdot}560$ (1), $b = 30{\cdot}962$ (4), $c = 9{\cdot}918$ (1) Å, $\beta = 91{\cdot}02$ (4)°, $Z = 4$, $D_x = 1{\cdot}618$ g cm^{-3}. The structure has been solved by direct methods and refined to $R = 0{\cdot}033$ for 3400 independent observed X-ray reflexions. Co is coordinated to six O atoms of water molecules which form a nearly regular octahedron. These octahedra are linked by hydrogen bonds to the hydrogen